Investigating physical and rheological properties of foamed bitumen

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1. Introduction

Keeping roads in good condition requires regular maintenance activities. This maintenance, given the large extent of the road networks, consumes a massive amount of non-renewable resources, mainly virgin aggregates. In addition, standard maintenance and rehabilitation activities create delays for users, traffic capacity deficiencies, safety issues for construction site workers and drivers, and demand a large amount of material handling and equipment. The economic crisis, increased costs of materials, and a strong desire to maintain a safe, efficient, and sustainable roadway system have fueled a resurgence of recycling existing pavement as a primary option.

It is acknowledged that limiting the disposal of old pavement materials, therefore minimizing the use and transport of virgin aggregates, as well as reducing landfilling, lowers environmental impact. These benefits, combined with the lower temperatures used in asphalt recycling, might lead to the belief that recycling always represents an eco-effective strategy. However, producing asphalt mixes at lower temperatures represents a successful alternative only if the final pavement is then able to compete, in terms of durability for instance, with standard hot-mixes. Also, the environmental effects of foaming agents or additives, if used, have to be as low as possible to achieve good results in the Life Cycle Assessment (LCA) of the product.

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Currently, different techniques are used to recycle existing pavements. One of the most adopted is based on foamed asphalt mixes. However, despite significant advancements in the last decade, the adoption of foamed asphalt still relies substantially on empirical trials and lacks universally accepted mix-design procedures. Foamed asphalt mixes are mainly adopted by European countries as a cost-effective replacement for base and sub-base layers; full-depth recycling and cold-in-place methodologies are generally considered hard to implement on upper surface layers due to high inhomogeneity of the existing paved materials (e.g., different age of the sections to be recycled, dimension and shape of aggregates, binder content, oxidation of the binder, etc.) even for relatively small areas. Moreover, it is generally agreed that cold-recycled mixes end up being a cost-effective method but with inferior long-term performance with respect to hot-mixes.

The mechanical performance and durability of foamed asphalt mixes rely on the physical and rheological properties of foamed bitumen, which should be in turn assessed. The present study investigated bitumen properties before and after being foamed. Besides traditional testing (penetration and Ring and Ball [R&B]) more advanced tests were conducted on foamed bitumen using the following equipment: Brookfield Rotational Viscometer, Dynamic Shear Rheometer (DSR), differential scanning calorimetry (DSC), and optical microscope imaging.

1.1. Background

Foamed bitumen is a mixture of hot bitumen, water, and air; sometimes a foaming additive is added to improve the quality and the stability of the foam. Spraying simultaneously hot bitumen (normally between 150 and 180 °C) and water (ambient temperature) causes the mix to expand several times its original volume generating a fine mist or foam.

The production of effective foamed bitumen mixes for pavement recycling applications can only be achieved when the bitumen adopted shows suitable foaming characteristics. Efficient methodologies for defining bitumen foaming properties are consequently of critical importance if efficient roadways are to be constructed or recycled. To date, foam is principally measured according to two main parameters: the expansion ratio (ER), which measures the increase of bitumen in volume after being sprayed, and the half-life (H-L), which evaluates the durability and the stability of the foamed-state before collapsing [1,2]. However, the adoption of ER and H-L has been criticized for several reasons [3–6]:

1. these parameters do not measure a physical material property, and any correlation between the test results and performance has to be ascertained through experience.
2. These parameters do not permit a full understanding of the mechanism of foamed asphalt and how it forms and decays; consequently, it is not understood why certain bitumens make good foam while others do not, even if very similar; and
3. the ER and H-L tests are operator-dependent and therefore highly variable.

Theoretically, the viscosity of bitumen is acknowledged as one of the main variables influencing the foaming properties; softer bitumen with a low viscosity produce higher expansion ratios and longer H-Ls with respect to harder, high-viscosity bitumens, whilst the use of high-viscosity bitumen is assumed to achieve superior coating of the aggregates [7]. However, the effect of viscosity on foaming potential is still not entirely clear.

Limited literature also exists on the effects of foaming processes on bitumen chemistry and, vice versa, how bitumen chemistry affects the potential foaming properties. Studies by Barinov [8] showed that increasing the asphaltene fractions of bitumen increased the ER and H-L; asphaltenes can indeed act as surfactants that delay the foam from collapsing.

Lesueur et al. [3], realized that bitumen composition did not greatly influence the foaming properties. Namutebi et al. [9] revealed that the presence of waxes within the binder enhances the foam characteristics. Jenkins [10] reported as a drawback that some bitumen may a priori contain anti-foam additives, incorporated during refining processes to precisely avoid the creation of foam during manufacturing and hauling.

In addition, investigating the foaming properties of bitumen before and after the foam has been created entails several complications. As said, foam remains stable for a relatively short amount of time (seconds) and then collapses; collecting samples in a foam-condition state and suddenly performing tests is therefore hard to achieve (i.e., if a standard penetration test is conducted at 25 °C after the sample has been cured in a constant temperature water bath for at least one and a half hours [11], the original foam will partially disappear in that time). If left at ambient temperature for a long time, foamed bitumen might lose part of its air content (bubbles). Sunarjono [12] tried to preserve the foam-condition state by exposing samples of foamed bitumen to very low temperatures in a refrigerator just after spraying in order to conduct testing later; Kutay et al. [13] soaked foamed bitumen samples in liquid nitrogen to instantly freeze the foam and conduct tests later.

The present study investigated the foaming properties of bitumens, correlating standard foam-experience results (ER_M and H-L) with rheological and chemical properties of the material before and after being foamed.

1.2. Objective

The objective of this paper was to evaluate the effect of foaming processes on physical, rheological, and chemical properties of three bitumens having the same penetration grade but sourced from different refineries. The effectiveness of a foaming additive was also investigated.

2. Materials and experimental program

2.1. Materials

Three types of bitumens were sourced from three different refineries in the northern part of Italy and examined further. For the purpose of the present research, they were named Bitumen A (Bit A), Bitumen B (Bit B), and Bitumen C (Bit C). Although coming from different refineries, they were classified as having the same penetration grade, 70–100 dmm [14].

A foaming additive was also added to Bit B and Bit C for analyzing its influence on bitumen properties before and after the foaming process. Bit B and Bit C blended with the foaming additive (ADD) are denoted as Bit B + ADD and Bit C + ADD. The specific density of the foam agent was 0.9 g/cm^3; the flash point was 170 °C; its chemical composition was based on oleic acid diethanolamine. The recommended dosage by the supplier ranged between 0.4% and 0.6% by weight of bitumen; in particular, the 0.6% concentration was used in the present study. The standard physical properties of the bitumens used are provided in Table 1.

It can be observed that (1) even if assessed as having the same penetration grade (70–100 dmm), Bitumen A exhibited a higher penetration, probably due to unidentified additives adopted for stabilizing bitumen during refining and hauling; (2) the foaming additive (Bitumens B + ADD and C + ADD) provided a consistent softening of neat Bitumen B and C, resulting in higher penetration and lower softening temperature. Table 1 also shows penetration indexes (PI) for unfoamed bitumens ranging between −0.68 and −1.91. Good paving bitumens usually provide a PI between −1 and +1, while temperature-susceptible bitumens present a PI lower than −2 [14]. Bitumen A was, therefore, the most temperature susceptible among bitumens tested.

The experimental plan included four main sections:

(a) evaluation of physical, rheological, and chemical properties of three types of virgin bitumens (a foaming additive was then added to two of them);
(b) assessment of the foaming properties using the Wirtgen Laboratory-Scale foamed bitumen machine (WLB-10S);
(c) characterization of physical and rheological properties of post-foam bitumens; and

Table 1
Penetration, R&B softening temperature and penetration index for unfoamed bitumen.

	Bitumen A	Bitumen B	Bitumen B + ADD	Bitumen C	Bitumen C + ADD
Penetration 25 °C, 100 g, 5 s (dmm)	133	75	103	86	130
Ring-and-ball softening point (°C)	40	46	45	47	45
Penetration index	−1.91	−1.37	−0.68	−0.61	0.1

(d) optical microscope imaging of foamed bitumen samples. Fig. 1 summarizes the experimental program.

2.2. Procedures

Bitumen foaming tests were conducted using the WLB-10S laboratory equipment to determine the optimum foaming water content and optimal foaming temperature according to the following test conditions:

- air pressure: 550 kPa;
- water pressure: 600 kPa;
- temperature of bitumen: 150 °C, 160 °C, 170 °C, and 180 °C;
- water content: 1%, 2%, 3%, and 4%;
- amount of bitumen being foamed during each test: 500 g at a rate of 100 g/s.

Wirtgen [1] recommends measuring two empirical indexes to determine the optimum foaming water content and temperature: (a) expansion ratio (ER) and half-life (H-L). Expansion ratio is defined as the maximum increase in volume due to the foam with respect to the original volume of bitumen; half-life is the time elapsed (seconds) for the foam to collapse to half its maximum volume. Maximum ER (ERM) is generally considered to be correlated to the viscosity of bitumen. On the other hand, H-L is a measure of stability of the foam and provides information about the collapse rate of the foam [1].

2.3. Bitumen sample collection

A specific protocol was designed to preserve the bitumen–air–water system that is supposed to form the foamed bitumen after foaming [10,12,13]. To this end, foamed bitumen samples were collected in different containers and then left for cooled for 15 min at room temperature. Afterward, the containers were placed in a climatic chamber at −10 °C until the testing day. By using this protocol the bitumen–air–water system of the collapsed foam was preserved by spontaneous curing processes such as water evaporation and air-releasing phenomena. This approach was also adopted in other research studies on foamed bitumens [12,13]. Standard penetration containers according to [11] were directly filled by pouring from the foaming bucket and stored at 25 °C until testing; penetration tests were performed within 4 h after foaming.

2.4. Rotational viscometer

Measurements of rotational (dynamic) viscosity were conducted using a Brookfield viscometer according to [15]. Viscosity tests were carried out at varying temperatures between 90 °C and 180 °C to test over the entire temperature domain occurring during foam production (150–180 °C) and foam-related properties assessment; the temperatures of foamed bitumen directly after spraying were measured in the 90 °C to −120 °C range.

2.5. Rheological analysis

Rheological characterization was conducted by using a Dynamic Shear Rheometer (DSR) in controlled strain mode with a parallel plate geometry system (25 mm in diameter) and a final gap of 1 mm [16]. The frequency was set at 10 rad/s (almost 1.6 Hz), and the temperature was varied starting from 46 °C with a 6 °C increment until the SuperPave original bitumen performance grade was determined [17].

2.6. DSC test and microscope images

Differential scanning calorimetry (DSC) was performed to investigate the thermal behavior of selected bitumens and the presence of possible waxes. Samples of 10 mg of unfoamed bitumens (Bitumens A, B, B + ADD, C, and C + ADD) were subjected to the same testing temperature interval ranging between –100 °C to +100 °C; samples were first cooled to −100 °C at 10 °C/min and then heated to 100 °C at 5 °C/min. Nitrogen was adopted as the purge gas at a flow rate of 50 mL/min.

In addition, samples of foamed bitumens were then analyzed in a laboratory through a low-powered microscope. As mentioned above, foamed samples were conserved at −10 °C in flexible metallic containers (250–300 mm in height); after that, selected samples were soaked in liquid nitrogen to further force a brittle breakage of the solid foamed bitumen. Bubble morphology and distribution were guaranteed by using this procedure. Optical evaluation by microscope had two main goals: (a) to determine the order of magnitude of the bubbles as a function of the foaming water content and foaming temperature, and b) to identify possible water entrapped in the foamed bitumen samples.

3. Results and analysis

3.1. Foaming characteristics: expansion ratio and half-life

Maximum expansion ratio (ER_M) and half-life (H-L) were determined for all bitumens. Four different foaming temperatures (150 °C, 160 °C, 170 °C, and 180 °C) and four foaming water contents (FWCs) (1%, 2%, 3%, and 4%) were tested.

ER_M and H-L were measured following the procedures recommended by Wirtgen [18]. H-L was determined as the time in

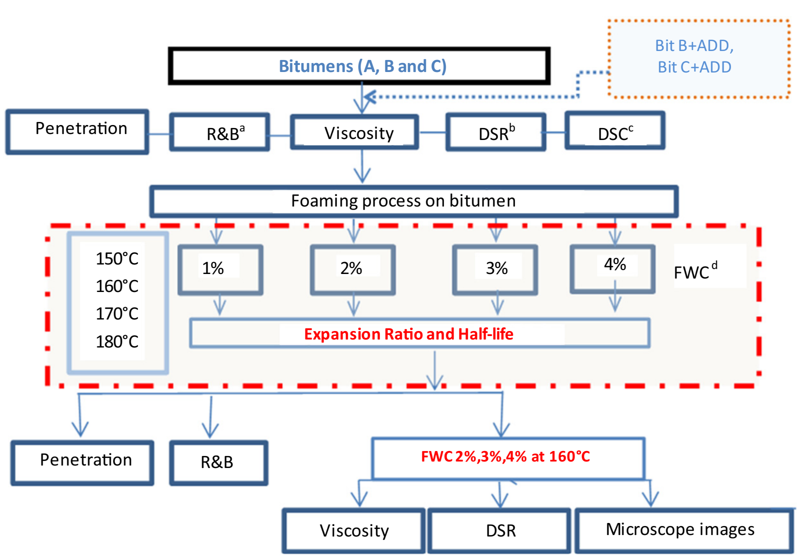

Fig. 1. The experimental program. [a]R&B: Ring and Ball temperature. [b]DSR: Dynamic Shear Rheometer. [c]DSC: differential scanning calorimetry. [d]FWC: foaming water content.

seconds that the foamed bitumen required to collapse from maximum expansion to half of maximum expansion.

Table 2 shows the foaming properties of the tested bitumens, expressed as mean values of three replicates and standard deviation. Bit A and Bit C proved to have better foamability than Bit B independent of temperature and FWC. The addition of the foaming additive (Bit B + ADD) provided Bit B with a longer H-L and ER_M, improving therefore the overall foaming capacity. Bit B was identified as providing poor foaming capacities, low expansion ratios (ER_M), and a very short H-L. On the other hand, Bit C exhibited the greatest values of foaming parameters among the bitumens without the addition of the foaming additive. It showed H-L values greater than 35 s for all temperatures and FWCs. It should be noted that Bit C provided very good foam characteristics, showing extremely high H-Ls and very good ER_M. These values were in the same order of magnitude as Bit B + ADD foaming characteristics.

The foam stability in Bit B + ADD, Bit C and Bit C + ADD, provided very high H-L values even exceeding two minutes for some particular temperatures and FWC; this could in turn be particularly helpful during the mixing stage by providing a better aggregate coating and mix workability [10,19–22]. In the case of Bit B + ADD, ER_M was also improved by the foaming additive doubling its expansion volume. It should further be noted that the foaming properties of Bit B were very poor and it did not reach the minimum $ER_M = 8$ and H-L = 6 s requirements [1,2] in most of the tested cases. Bit C performed generally better than both Bit A and Bit B, being similar, in terms of the H-L, to bitumens with foam additive (ADD). Differences among bitumens, even if having the same consistency grade, proved that extraction and refinery processes cause important variations in the final performance of bitumen. Also, high-penetration bitumens (softer) showed a better foam-ability relative to harder bitumens although a strict correlation cannot be rigorously assessed.

FWC influenced equally and independently from sourcing or use of additives, the higher the FWC the higher the ER for all temperatures; similar results have been confirmed in the literature [4,9,21,23]. However, increasing the foaming temperature did not always provide better ERs and H-Ls for the same FWC (e.g., Bit A at 150 °C and FWC 4% had an $ER_M = 28$ while at 180 °C and the same FWC had an $ER_M = 22$; Bit B at 160 °C and FWC 4% had an $ER_M = 23$ while at 180 °C and the same FWC had an $ER_M = 14$). The results controvert some trends reported by several authors which agreed that increasing the foaming temperature had the effect of increasing the expansion ratio and decreasing the H-L [3,20]. Others noted that the rate of increase is reduced significantly when the temperature is increased from 170 °C to 180 °C [21].

FWCs between 2% and 3% proved to be the optimal equilibrium points in balancing an acceptable ER and H-L, thus confirming previous studies: Brennen et al.: FWC 2% at 160 °C [20]; Nataatmadja: FWC 2% to 2.5% at 160 °C [22]; Mohammad et al.: FWC 2.75% at 160 °C [23]; and Marquis: FWC 3% at 160 °C [24] (Fig. 2a and b).

On the other hand, higher ER_M's were observed at 150 °C and 160 °C for all bitumens while longer H-Ls were provided at 160 °C and 170 °C. Bit C and Bit C + ADD showed the best H-Ls at FWC 3% independently of the temperature. The highest ER_M's overall were experienced at 150 °C and 160 °C for all bitumens. The ER and H-L values are compared in Fig. 2 for a constant temperature (160 °C). It can be observed from Fig. 2 that the foaming additive significantly improved ER and HL for Bit B, making it suitable for foaming practices according to [1,2]. Bit C, which also had very good foaming characteristics, showed very small improvements due to the ADD in terms of ER but substantial enhancements in H-L values. Except for Bit B, all other bitumens exhibited good to very good foaming parameters when FWC was equal to or above 2%. Moreover, in analyzing Bit B + ADD and Bit C + ADD, 3% FWC

Table 2
Foaming properties of foamed bitumens.

	FWC (%)	Bit A				Bit B				Bit B + Add				Bit C				Bit C + Add			
		ER	SD[a]	H-L	SD	ER	SD	H-L	SD	ER	SD	H-L	SD	ER	SD	H-L	SD	ER	SD	H-L	SD
150 °C	1	9	0.6	23	2.9	7	1.2	6	1.2	10	2.8	58	0.7	8	0.0	72	3.5	10	1.4	180	0.0
	2	21	3.1	11	1.2	13	1.2	3	0.9	18	0.0	50	0.0	20	0.0	42	2.1	21	1.4	105	21.2
	3	26	2.8	8	0.7	16	2.5	3	0.3	26	2.1	91	15.6	27	1.4	44	5.7	32	0.0	70	14.1
	4	28	2.1	5	0.9	21	1.2	3	0.4	31	1.4	80	9.9	34	2.8	35	0.7	32	0.0	60	0.0
160 °C	1	8	0.0	35	0.7	6	0.3	9	1.5	10	0.0	62	2.1	9	1.4	90	0.0	6	0.0	180	0.0
	2	17	3.1	15	1.7	9	1.2	4	2.1	18	0.0	66	8.5	17	1.4	49	2.1	18	0.0	138	3.5
	3	23	3.1	10	0.6	13	3.5	2	0.1	25	4.2	95	7.1	26	0.7	57	0.7	26	0.0	153	17.7
	4	27	1.2	7	1.0	23	1.2	2	0.3	30	0.0	45	3.5	32	0.0	45	7.1	33	1.4	101	14.8
170 °C	1	6	0.0	31	1.4	5	0.7	8	1.8	7	1.4	51	0.7	6	0.0	61	15.6	8	0.0	185	0.0
	2	13	0.6	22	1.3	12	0.0	3	0.5	17	2.1	91	21.2	16	0.0	46	4.2	17	2.1	145	14.8
	3	18	0.0	11	1.4	11	1.2	3	0.3	23	1.4	130	42.4	22	0.0	60	5.7	22	2.8	210	0.0
	4	18	1.0	11	1.6	12	2.0	2	0.2	27	1.4	105	49.5	25	7.1	42	17.0	24	1.4	150	3.5
180 °C	1	10	0.0	23	0.6	5	0.0	12	0.6	5	0.0	75	7.8	4	1.4	85	7.1	6	0.0	135	21.2
	2	11	1.0	22	0.6	9	1.5	3	0.6	14	2.8	95	7.1	12	0.0	150	42.4	12	0.0	215	14.8
	3	14	1.5	18	0.6	10	1.5	2	0.3	20	2.8	120	0.0	20	0.0	125	7.1	19	1.4	188	3.5
	4	22	0.0	8	1.5	14	1.5	2	0.2	21	1.4	113	24.0	26	0.0	96	8.5	23	1.4	169	15.6

ER: expansion ratio in times.
H-L: half-life in seconds.
[a] SD: Standard deviation.

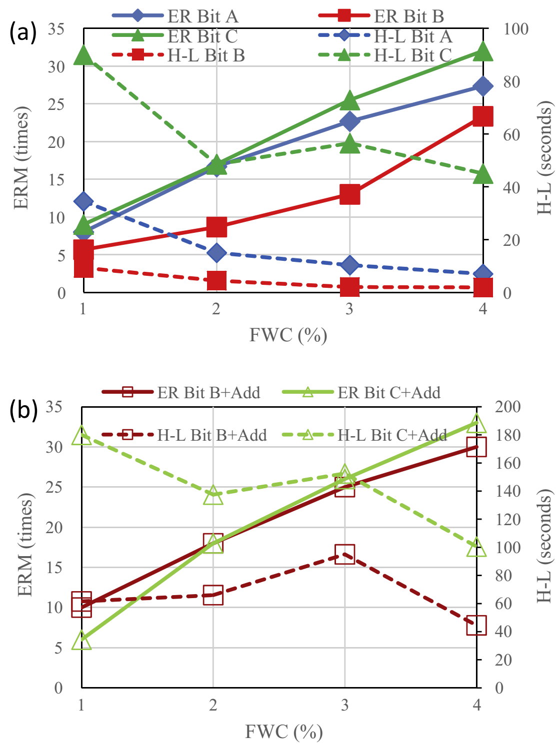

Fig. 2. Foaming characteristics of bitumens at 160 °C and FWC 1%, 2%, 3%, 4%. (a) Bitumens without ADD and (b) bitumens with ADD.

seemed to achieve the optimal H-L values, producing peak values for Bit C, Bit B + ADD, and Bit C + ADD. FWC 1% provided very small H-L and ER values regardless of the temperature effect.

3.2. Properties of foamed bitumens after foam collapse

As already mentioned, dealing with foamed collapsed bitumen is a hard task due to the several and fast modifications that occur in the foam immediately after spraying. In addition, limited research has been conducted on foamed collapsed bitumen; thus several technical and scientific questions remain without a robust answer: Will the physical and rheological properties of foamed bitumen be comparable to those of the original bitumen? Is there any water or vapor diffusion entrapped in the foamed bitumen? Physical, rheological, chemical (DSC), and optical (microscope images) evidences were therefore analyzed in an effort to answer these questions.

3.3. Physical characterization of foamed collapsed bitumen

Table 3 shows the penetration values of foamed bitumen samples for different foaming temperatures and FWCs. Fig. 3 exhibits penetration values in bar graph form for bitumens with FWC 1% and 4% at selected temperatures for comparative purposes. Error bars represent standard deviation. Penetration values after foaming exhibited very high variability throughout the sample; entrapped air within the foamed bitumen provided greater penetration if the needle passed through bubbles. It was consequently clear that such an empirical test was not appropriate to characterize foamed bitumen's properties. However, some range of magnitude can still be identified among the different sources of bitumen. Bitumen B, for instance, showed the greatest homogeneity in penetration distribution, providing the lowest standard deviation (SD). It should be noted that foamed Bit B, Bit C, and Bit C + ADD provided penetration values (when foamed at 180 °C) evidently smaller than penetration values at other foaming temperatures. This could be considered an effect of the possible aging of the bitumen due to high-temperature spraying.

Softening point temperature was also evaluated to quantify any visible effect of the foaming process and foaming additives on bitumen consistency. Fig. 4 shows R&B test results for all the bitumens, Error bars represent standard deviation; four foaming temperatures and two FWCs were considered. However, very small changes were observed in consistency (R&B temperature); Bit B softening temperature, for instance, only increased by 2 °C on average for 150 °C and 160 °C foaming temperatures and FWC 1%. The addition of the foaming additive did not provide significant modifications in bitumen consistency. Bit A also evidenced an increase in the R&B values independently of the FWC; the greatest increase was 4 °C after foaming with respect to the unfoamed Bit A.

3.4. Foaming effects on viscosity

Rotational viscosity permitted a more precise analysis of the changes in foamed bitumen's consistency by means of shear stress through a defined temperature domain. In the present study, the dynamic viscosity between 90 °C and 180 °C was evaluated. The first value (90 °C) represented the average minimum temperature of bitumen just after being foamed (in the bucket), and 180 °C was the higher foaming temperature. Viscosity and bitumen consistency have been correlated in several research studies [3,4,9,10,12], but several uncertainties still remain about the role of the consistency, viscosity, and bitumen surface tension in producing "good quality-foam." When analyzing foamed bitumen samples produced with different FWCs, some uncertainty still exists about the retained water after the foaming process. In this complex process, the very low percentage of water (ratio in weight of water to bitumen), works to interchange the heat transferred from bitumen to the water in the expansion chamber, exceeding the latent heat of steam inside the expansion chamber. A great part of this is evaporated, and just a very small quantity may remain entrapped within the bitumen–air–water system. Measurements to determine the mass loss at room temperature (25 °C) were undertaken on different samples of foamed bitumen without finding any significant variation at the 1/100-of-a-gram scale in the mass after 1 month.

Fig. 5(a–f) plots viscosity values for unfoamed and foamed bitumens at 160 °C (foaming temperature) and three FWCs (2%, 3%, and 4%). Fig. 5a depicts the viscosity of unfoamed bitumens. The effect of the additive can be also observed in Bit B + ADD and Bit C + ADD. The additive generally decreased the viscosity of Bit B (Bit B + ADD); viscosity was reduced by 10% at 135 °C and by almost 28% at 160 °C. Bit C appears to be insensitive to the effect of the additive (Bit C + ADD); no evident changes in the consistency were spotted. Therefore, it can be postulated that the effect of the additive on the bitumen's viscosity may depend on the type and source of the specific bitumen and its effect is independent of the bitumeńs initial penetration grade.

Considering bitumens without an additive and matching the outcomes from viscosity, penetration, and R&B tests, it is suggested that low-viscosity or low-consistency bitumens generally provided better foam-ability (Table 2, Fig. 2). Fig. 5b–f portray the viscosity values for foamed bitumens at three FWC values. In Fig. 5b, it can be seen that the viscosity in Bit A shows an increasing trend for higher FWCs. In fact, the viscosities for Bit A at FWC 3% and 4% were higher than Bit A at FWC 2%. Fig. 5c and d depict the effect of the foaming process on Bit B and Bit B + ADD. A very small increase in the viscosity for Bit B at FWC 3% can be minimally observed for all the temperatures tested. Notwithstanding, overall the foaming process appeared not to significantly affect the viscosity for Bit B.

Table 3
Penetration results of foamed collapsed bitumen.

Virgin		Foaming temperature (°C)							
Pen (dmm)		150		160		170		180	
	FWC (%)	Pen (dmm)	SD[a]	Pen (dmm)	SD	Pen (dmm)	SD	Pen (dmm)	SD
Bitumen A									
133	1	122	14.8	134	13.6	143.6	29.6	133.3	9.4
	2	120	5.0	125	9.6	89.0	5.4	146.5	8.9
	3	119	10.3	124	1.7	109.7	1.4	122.3	5.2
	4	132	0.6	113	11.7	119.3	19.3	135.8	5.3
Bitumen B									
75	1	92.3	4.0	96.7	2.5	95.0	22.6	74	3.7
	2	83.0	8.3	100.0	6.1	79.4	28.6	79	4.2
	3	86.3	4.9	106.5	8.5	96.0	4.8	75	6.1
	4	82.3	4.4	87.5	13.9	91.8	4.6	81	9.1
Bitumen B + ADD									
103	1	90	8.9	74	14.5	90.8	22.2	129.5	20.9
	2	101	21.6	74	24.8	86.3	17.8	132.2	19.9
	3	76	25.9	136	31.3	129.0	47.1	79.2	17.3
	4	67	8.2	113	39.5	94.2	11.3	94.8	22.3
Bitumen C									
86	1	109	26	114	38	95	8	68	13
	2	101	13	53	31	101	31	71	9
	3	88	49	102	23	72	25	62	12
	4	99	37	78	24	109	18	62	12
Bitumen C + ADD									
130	1	116	25	96	5	72	5	46	8
	2	90	34	103	7	79	11	64	15
	3	80	12	76	10	77	8	54	16
	4	100	26	94	14	63	10	67	21

Note: penetration values shown are the mean of at least 3 readings.
[a] SD: standard deviation.

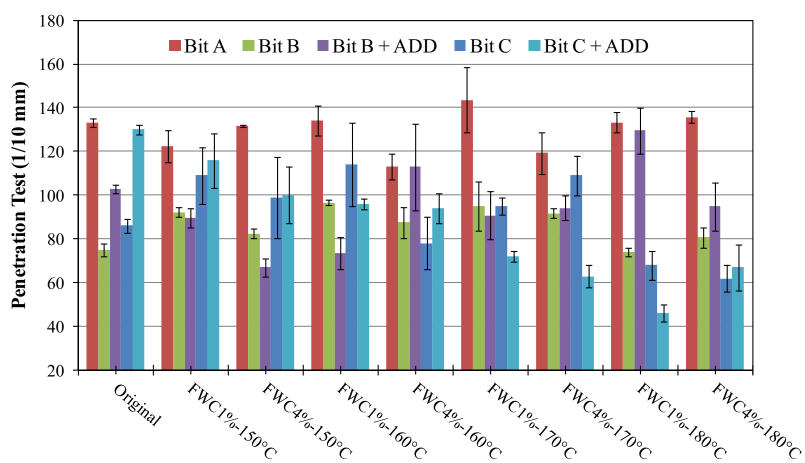

Fig. 3. Foaming influence on penetration.

Fig. 5e and f show the FWC effect on both Bit C and Bit C + ADD. Bit C showed an apparent increase in the viscosity for all the FWCs through the entire temperature range. Some trends can also be visualized below 130 °C; the higher the FWC, the higher the viscosity. However, this trend was not evident above 130 °C. For Bit C + ADD at FWC 2%, the increase in consistency is perceived at temperatures over 130 °C.

In analyzing bitumen without ADD, one can observe that Bit A and Bit C (softer bitumen) had greater foamability compared to Bit B (harder bitumen). However, this is just a general trend since Bit A (highest penetration values, lowest softening point, and lowest viscosity) did not exhibit the best foamability.

3.5. Foaming process effect on rheological behavior

Fig. 6 describes the rheological bitumen properties for all bitumens (unfoamed and foamed). Bitumen complex modulus (G^*) and phase angle (δ) were plotted as a function of the temperature for the different bitumens. Fig. 6a shows unfoamed bitumens, which experienced, as expected, the same viscosity trends (Fig. 5a). For instance, the phase angle δ, known as a viscous-elastic indicator, identified unfoamed Bit A as the most viscous and Bit B as the less viscous; Bit C provided a medium-to-low shear modulus trend. Bit A had the lowest G^* value and the highest phase angle δ, indicating a lower shear resistance and more viscous bitumen behavior,

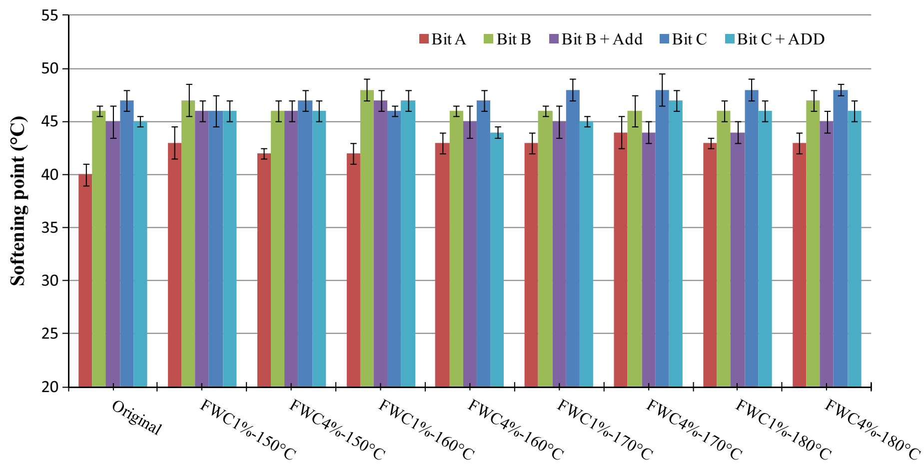

Fig. 4. Foaming influence on Ring and Ball softening point.

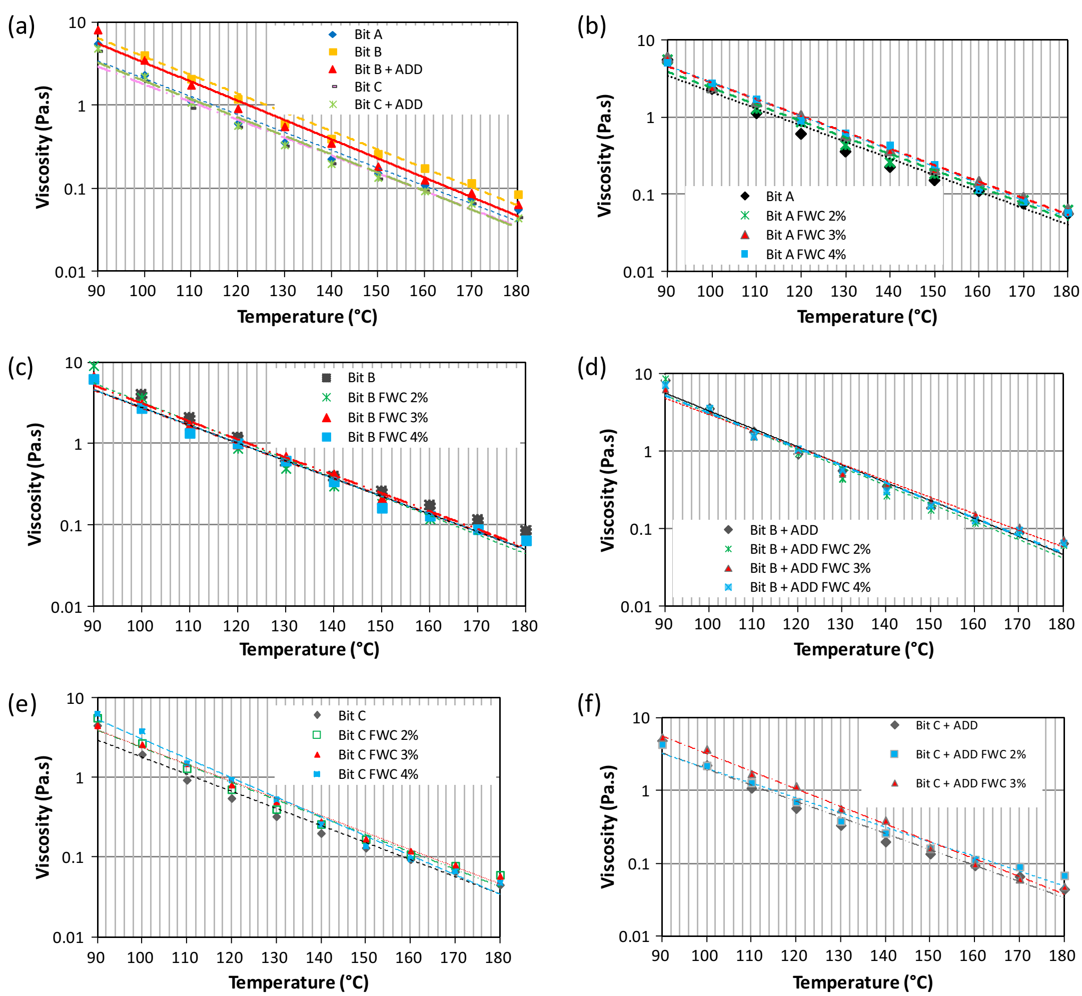

Fig. 5. Effect of foaming process on viscosity: (a) virgin (unfoamed) bitumens; (b) Bit A; (c) Bit B; and (d) Bit B + ADD, (e) Bit C, (f) Bit C + ADD.

making it more susceptible to permanent deformation at high service temperatures. Similarly to viscosity, the complex modulus for Bit B + ADD and Bit C + ADD underwent a decrease in the complex modulus, being more evident for Bit B than for Bit C; the G^* modulus for Bit B + ADD diminished by almost 30% (i.e., from 13 kPa to 10 kPa at 46 °C). For Bit C + ADD, the complex modulus diminished around 8% (i.e., from 7.8 kPa to 7.3 kPa at 46 °C).

Fig. 6b illustrates the foaming effects on the rheological behavior of Bit A. A slight softening was noted after the foaming process relative to the unfoamed condition, especially at lower temperatures; however, both G^* and δ were almost uniform throughout this temperature range. However, the complex modulus was insensitive to FWC above 52 °C, thus showing a unique trend. Phase angle exhibited similar insensitivity to FWC.

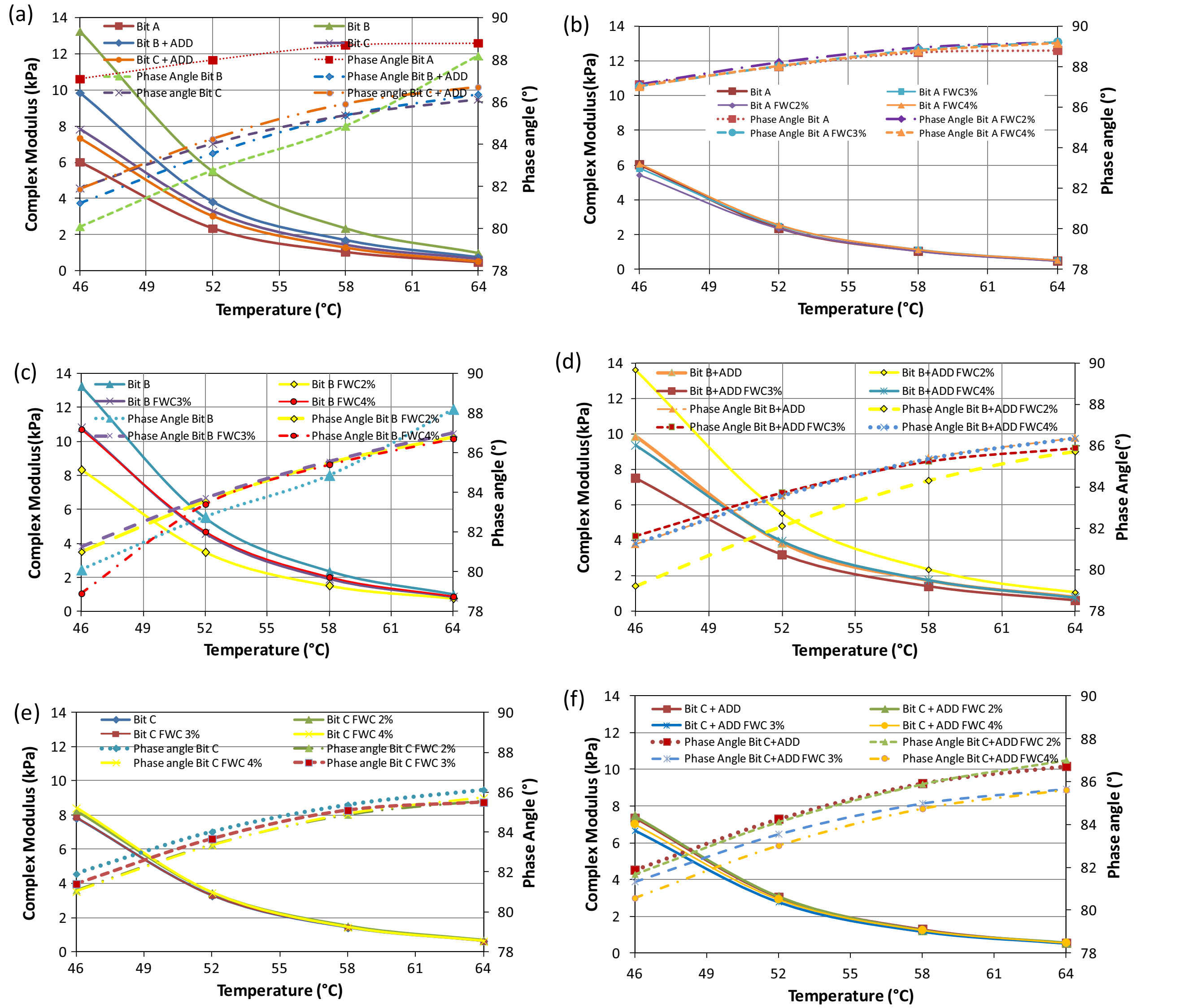

Fig. 6. Effect of foaming processes on rheological properties [foaming temperature 160 °C] (a) virgin (unfoamed) bitumens; (b) Bit A; (c) Bit B; and (d) Bit B + ADD, (e) Bit C, and (f) Bit C + ADD.

Fig. 6c illustrates the effect of the foaming process and the foaming water effect on the rheological behavior of Bit B. Again, similarly to Bit A, Bit B showed some kind of softening due to the foaming process; G^* modulus was on average reduced by the foaming process from 37% to 18% at 46 °C and 58 °C, respectively, depending on the FWC.

The phase angles δ increased in correspondence with G^* with respect to the unfoamed bitumen showing that the bitumen softens toward a more viscous material after the foaming process. This behavior was evident for temperatures greater than 49 °C; on the other hand, an unclear trend was noted for lower temperatures among the different FWCs. Fig. 6d portrays the effect of the additive on Bit B (Bit B + ADD). The effect of the additive together with the foaming process provided a 30% hardening for the sample at FWC 2% with respect to the unfoamed bitumen. This effect was also tangible in the decrease of the phase angle δ over the entire temperature range. For the other FWCs, the effect is varied. For instance, Bit B + ADD FWC 4% exhibited a stiffness very close to unfoamed bitumen, whereas for Bit B + ADD FWC 3% a noticeable reduction was verified for all the temperatures tested.

Fig. 6e and f show the effect of the foaming process in the rheological behavior for Bit C and Bit C + ADD. Complex moduli for both samples did not reveal significant changes after the foaming process; very few differences could be identified below 52 °C. Bit C without the additive (Fig. 5e) exhibited an increase in G^* modulus only perceptible for FWC 2% and 4%. Instead, Bit C + ADD suffered some softening for higher FWCs. When analyzing δ behavior, changes were more evident and consistent along the temperature domain. In regard to Bit C, the foaming process reduced the phase angle, improving the elastic response of Bit C. The trend appeared to be related to higher FWCs, but this trend is not consistent in the case of FWC 2%. Due to the strong inhomogeneity of the material and the very little amount of bitumen tested during each DSR test, some variability in the results was experienced and a higher number of samples is recommended in future research.

Rheological analysis on foamed bitumens revealed different behavior depending on bitumen type. Bit A and Bit C had good to very good foamability, exhibiting almost no effect in their complex modulus behavior, while the phase angle was more sensitive to the FWC. Bit A showed slight changes in both complex modulus G^* and phase angle δ. Bit C and Bit C + ADD exhibited more noticeable changes in the phase angle δ, revealing a clearer trend: the higher the FWC, the lesser the phase angle. Bit B, acknowledged as a bitumen with very poor foamability, exhibited an evident sensitivity to FWC in both complex modulus and phase angle evaluations.

3.6. Microscope images and DSC

Fig. 7 illustrates selected microscope images of foamed Bitumen B + ADD at two foaming temperatures and two FWCs; samples were rapidly frozen after foaming by using liquid nitrogen to preserve the internal bubble distribution and morphology. The microscope analysis graphically confirmed the difference between higher and lower FWC. As well documented in recent studies [9,10], a greater FWC increases the ER; this can be visually confirmed by images of lower FWC (1%) samples that exhibit bubble dimensions significantly smaller than higher FWC (4%) samples, independent of the foaming temperature. Bubble diameters were roughly evaluated to be between 170 μm and 330 μm for FWC = 1%, whereas they increased to the range of 600 μm to 1000 for FWC = 4%. In [13], diameters of bubbles for different foamed bitumens were reported with a similar order of magnitude. There is no statistical evidence on the exact diameter of the bubbles since different results could be obtained by sampling the bubbles in a position that does not pass for the center of the bubble (smaller diameter), for instance. However, the results seemed to be consistent with other previous studies [13]. The qualitative evaluation based on the measurements conducted, resulted useful to verify roughly the magnitude range of bubbles in bitumens.

DSC is acknowledged in the literature for providing important thermal parameters such as the bitumen glass transition temperature (T_g), the melting temperature range of crystallites, and the enthalpy of melting [24,25]. In addition, it has been useful for studying the presence of wax in bitumen [26,27,28]. By using DSC, a previous study found that the presence of waxes in bitumen accounted for improvements in foaming characteristics [9]. DSC scans were applied to unfoamed bitumen samples to investigate thermal behavior, wax presence, and the effect of the additive on the bitumen thermal properties. To reset previous thermal bitumen history, samples were heated from room temperature to 100 °C during 2 h, and then cooled at room temperature for 24 h. DSC thermographs for the different bitumens are displayed in Fig. 8. Thermal properties were analyzed according to the ASTM 3418-08 [29]. Fig. 8, shows schematically how T_g were determined. T_g temperatures ranges were between −40 °C and −42 °C except for Bit A which yielded a T_g at −22 °C. Additive seemed does not affect significantly T_g for Bit B and Bit C. When the glass transition region is achieved, bitumen is in a metastable state causing the material to continuously shrink isothermally [25,26]. The figure, shows that for Bit A, Bit B + ADD, Bit C, and Bit C + ADD one clear exotherm peak located very close to 30 °C, which is a indicative of a crystallization phase (crystalline wax) [27]. Following the procedure mentioned in [27] the endotherm area can be also identified (e.g., for Bit C between −16 and 73 °C). The endotherm area can determined by integrating the area between the transition zone and a straight line extrapolated from baseline above 73 °C of the thermograph. Such area is directly proportional to the crystalline wax content [9,27,28]. Similar results, but in very limited bitumen samples were found by [9], in bitumens with good foam-

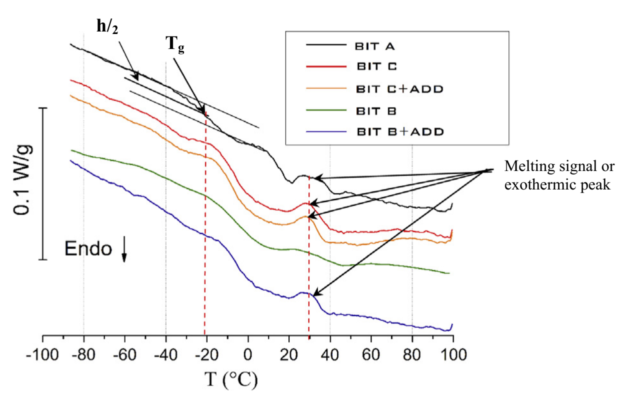

Fig. 8. Differential scanning calorimetry of bitumen tested.

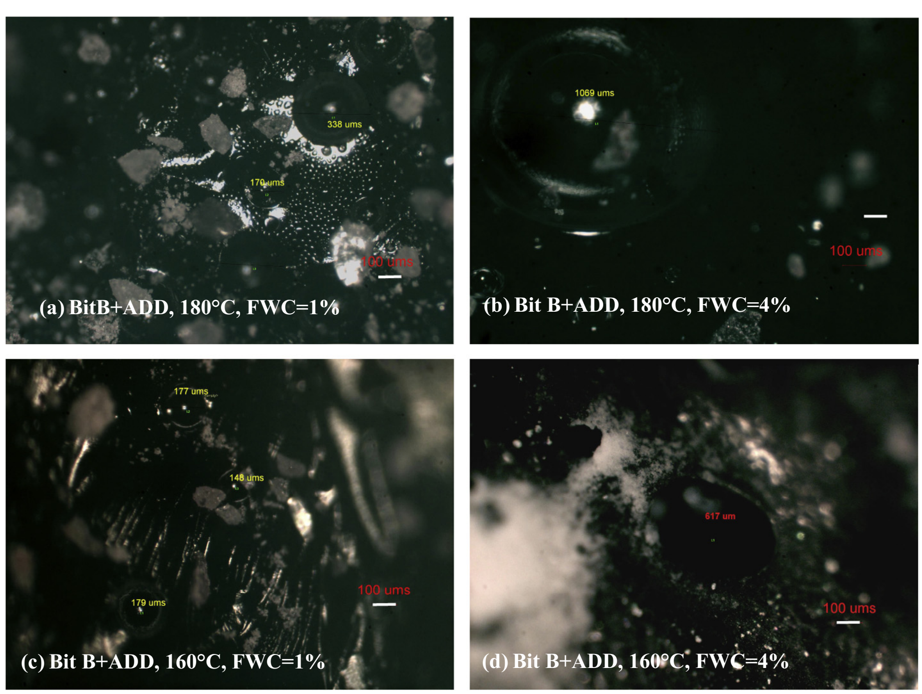

Fig. 7. Internal morphology of foamed bitumen with foaming additive (a) foaming temperature (FT) 180 °C, FWC = 1%; (b) FT 180 °C, FWC = 4%; (c) FT 160 °C, FWC = 1%; (d) FT 160 °C, FWC = 4%. Note: *Optical microscope enlargement was* 50 *X.*

ing properties by using DSC analysis. The foamability of bitumens studied matched with the findings from thermal properties. Bitumen exhibiting well to very well foaming properties performed visible crystallization phases (Bit A, Bit B + ADD, Bit C and Bit C + ADD). Conversely, Bit B, which did not perform an exothermic peak provided poor foaming properties.

4. Discussion

Foaming process effects were studied on three standard bitumens, as well as two other bitumen samples modified with the addition of an foaming additive for improving their foamability (Bit B + ADD and Bit C + ADD). Classical standard tests (penetration, R&B temperature) were conducted together with rheological characterizations (viscosity, DSR). Classical tests, in general, revealed some insights about the consistency of the foamed bitumens. However, the penetration test demonstrated its inappropriateness for describing the behavior of such a complex material. R&B temperature, instead, showed more consistent results but still was not able to clearly define the material properties. Viscosity and DSR measurements provided a better insight and proved to be more appropriate for macroscopic evaluation of foamed bitumens. Results from the viscosity test on foamed bitumen reflected more visible changes as effects of the foaming process; indeed, such changes were more evident on Bit A and Bit C, bitumens with good foamability. On the other hand, Bit B, which provided limited foamability, exhibited minimal changes in this kind of test. In contrast, results obtained with the rheometer on bitumen with good foamability exhibited little sensitivity to the FWC.

As a clear trend showing the effects of FWC on foamed bitumen has not been identified in the literature [12,30,31], it is postulated that rotational viscosity may be better able to identify the FWC effect of the foaming process on bitumens with good foaming properties. The findings in the present research agreed with the behavior found by [30], in the sense that the foaming process influences the elastic response of foamed bitumens.

DSR and viscosity tests confirmed that the foaming additive altered the consistency of the tested bitumen before foaming. The results of the analysis also revealed that the properties of bitumen changed after the foaming process and that such a change might be a function of the curing time (moisture dissipation rate of the binders). The diffusion of water vapor as reported by [32] has not received much study and various values are reported in the literature (Nguyen et al. [33]: diffusion coefficient [DC] of 5.04×10^{-5} mm^2/h). It is suggested that the foaming properties of bitumen have to be assessed by considering more thermodynamic parameters, for instance, water vapor diffusion coefficients.

Microscope image analysis also showed the relationship between the moisture content (FWC) and bubble dimension. The results suggest that the freezing method was useful in preserving foam-condition samples.

5. Conclusions

An experimental program was conducted to evaluate the physical (penetration test, R&B softening point), rheological, and foamability properties of two bitumens sourced from different refineries but having the same penetration index. The following conclusions can be drawn:

- foaming evaluation showed that Bitumen A had a good foamability, and Bitumen B had a limited foaming capacity; however, the foaming additive (Bitumen B + ADD) significantly improved the foaming properties (more than 10 times in H-L).
- Physical properties by means of penetration tests and R&B temperature were not considered appropriate for foamed bitumens. Bitumen B did not exhibit considerable changes in R&B values. Bitumen B + ADD showed a slight decrease in R&B for foaming temperatures higher than 160 °C, temperatures decreased between 2 °C and 4 °C.
- Bubble morphology assessed through the microscope showed a correspondence between bubble magnitude and higher FWC for both temperatures analyzed, and no water presence was detected.
- The addition of a foaming additive marginally decreased viscosity (maximum reduction was 20%). In general, after foaming, a softening behavior was observed, with the magnitude depending on the FWC.
- Outcomes pointed out that physical evaluation did not determine a direct relationship with foaming performance. While more fundamental tests such as viscosity and DSC, revealed better understanding and correspondence with foaming properties. A simultaneous chemical investigation regarding bitumen composition and source should be also conducted.

Numerical simulations of the different thermodynamical variables and bitumen properties might explain some foaming properties of bitumens, thus avoiding long empirical campaigns. Finally, more practical and rational methods should be developed for assessing foaming properties and reducing subjectivity and operator dependence.

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